

PATENT
CASE 4233C3

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicants: Donald B. Appleby et al : Group Art Unit: 1211
Serial No.: 08/360,184 : Examiner: E. White
Filed: December 20, 1994 :
For: Polyol Polyester Synthesis

DECLARATION UNDER 37 C.F.R. §1.608 OF K. MICHAEL WIESER

Assistant Commissioner for Patents
Washington, DC 20231

Dear Sir:

I, K. MICHAEL WIESER, declare that:

1. I am employed by the assignee of the present application, The Procter & Gamble Company, and have been working for The Procter & Gamble Company continuously since 1966.
2. From 1985 to 1990 and 1995 to 1997 I worked as a technician on the sucrose polyester synthesis project in which sucrose and fatty acid methyl ester were reacted to form sucrose fatty acid esters, commonly referred to as sucrose polyesters. I was responsible for overseeing preparation of reactant feed batches for pilot plant continuous sucrose polyester production processes

and for analyzing samples of reactor streams from pilot plant continuous sucrose polyester production processes according to established procedures.

3. I participated in the pilot plant continuous sucrose polyester production process designated P90117 which was conducted from January 18, 1989 to January 27, 1989. The P90117 pilot plant continuous sucrose polyester production process, including operation of the continuous sucrose polyester synthesis equipment, sample collection and data preparation, was performed under the direction and control of Mr. Scott Pearson.

4. Under my supervision and control on January 17, 1989, a sucrose feed batch for the P90117 pilot plant continuous process comprising sucrose, fatty acid methyl ester and soap was prepared and a catalyst batch for the P90117 pilot plant continuous process comprising potassium carbonate and fatty acid methyl ester was prepared. I have reviewed Exhibits 3 and 4 and confirm that they comprise accurate copies of the control records evidencing the components employed in forming the sucrose feed batch and the catalyst batch, respectively, for the P90117 pilot plant continuous process. Exhibits 3 and 4 bear my signature indicating that I oversaw the preparation of the respective batch compositions. To prepare the sucrose feed batch, potassium soap was prepared in a reactor 001 by a reaction of about 600 pounds of fatty acid ester (I-1) and about 115 pounds of potassium hydroxide in the presence of about 1200 pounds methanol, as set forth in Exhibit 3, the methanol was evaporated off, and about 3700 pounds of fatty acid methyl ester (ester blend Y) and about 800 pounds of sucrose were added, as also set forth in Exhibit 3. To prepare the catalyst batch, about 250 pounds of potassium carbonate and about 750 pounds of fatty acid methyl ester (ester

blend Y) were mixed in reactor 501, as set forth in Exhibit 4. Both the sucrose feed batch and the catalyst batch were substantially solvent free.

5. During the P90117 pilot plant continuous sucrose polyester production process, I also determined the weight percentages of potassium carbonate catalyst (as carbonate) and potassium soap (as soap) in samples taken from the recirculation loop of the initial reactor stage, i.e., the first reactor R600, according to established procedures described below, and I accurately recorded the determined weight percentages of potassium carbonate catalyst and potassium soap in the samples on data sheets which were maintained in the pilot plant for the purpose of recording such data. The data which I accurately recorded included the date and time at which a sample was taken, the reactor recirculation loop from which the sample was taken, and the determined weight percentages of potassium carbonate catalyst and potassium soap in the sample. I also initialed the entry for each sample which I analyzed.

6. I have examined Exhibit 7, and I confirm that Exhibit 7 comprises accurate copies of the data sheets from the P90117 pilot plant continuous sucrose polyester process on which I accurately recorded the date and time at which a sample was taken, the reactor recirculation loop from which the sample was taken, and the determined weight percentages of potassium carbonate catalyst (as carbonate) and potassium soap (as soap) in the sample. The entries bearing my initials (KMW) indicate those samples which I analyzed as described herein and for which I recorded the data as described herein.

7. The established procedure for determining the weight percentages of potassium carbonate catalyst (as carbonate) and potassium soap (as soap) in a sample during the P90117 pilot plant continuous process comprised titration with a 0.1 Normal HCl solution utilizing a Kyoto At-118 microprocessor-controlled automatic titrator. A weighed reactor sample was mixed with a neutral solution comprising equal parts of isopropanol and water. The sample mixture was heated with stirring until the temperature reached about 100°F and then was heated for about 2 minutes at about 100°F or until the sample dispersed completely. The sample mixture was placed on the titrator and was automatically titrated until the pH reached 3 or below. The automatic titration indicated that there were two equivalence points. The first equivalence point corresponded to the stronger base, the potassium carbonate catalyst (as carbonate), and second equivalence point corresponded to the weaker base, the potassium soap (as soap). The weight percentages of potassium carbonate catalyst (% Carbonate) and potassium soap (% Soap) in the sample were calculated from the titration volumes needed to reach the two equivalence points, respectively, using the following equations:

$$\% \text{ Carbonate} = \frac{\text{EP1 (Vol)} \times \text{Normality HCl} \times 13.8}{\text{Sample Wt (g)}}$$

$$\% \text{ Soap} = \frac{[\text{EP2 (Vol)} - 2 \times \text{EP1 (Vol)}] \times \text{Normality HCl} \times 32.0}{\text{Sample Wt (g)}}$$

wherein EP1 was the volume of the HCl solution need to reach the first equivalence point, EP2 was the volume of the HCl solution need to reach the second equivalence point, and Normality HCl was the Normality of the HCl titration solution, as set forth on the third page of Exhibit 7.

8. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these

statements were made with knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Respectfully submitted,

By: K. Michael Wieser
K. Michael Wieser

Date: 4-5-99

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